

**TOTAL AND AMENABLE CYANIDE: DISTILLATION**  
**EPA 9010C Revision 3 (November 2004)**

Facility Name: \_\_\_\_\_ VELAP ID: \_\_\_\_\_

Assessor Name: \_\_\_\_\_ Analyst Name: \_\_\_\_\_ Inspection Date: \_\_\_\_\_

Relevant Aspect of Standards	Method Reference	Y	N	N/A	Comments
Records Examined: SOP Number/ Revision/ Date _____ Analyst: _____					
Sample ID: _____ Date of Sample Preparation: _____ Date of Analysis: _____					
1) For nonpotable water, were samples cooled, $\leq 6^{\circ}\text{C}$ , adjusted to pH $>10$ with NaOH, and reducing agent added if oxidizer present (such as sodium thiosulfate if residual chlorine is present or $\text{H}_2\text{O}_2$ if sulfur compounds are present)?	40CFR136.3 Table 1I				
2) For drinking water, were samples cooled, $\leq 6^{\circ}\text{C}$ and adjusted to pH $>12$ with NaOH?	40 CFR 141.23 k(2), EPA 815-R-05-004				
3) For nonpotable water or drinking water, were samples analyzed within 14 days?	40 CFR 136.3 Table 1I, 141.23 k(2)				
3) Are samples checked for presence of oxidizing agents prior to distillation by testing a drop of sample with potassium-iodide starch test paper? ( <i>Blue color indicates a need for treatment.</i> )	6.2				
4) If treatment is required for oxidizing agents, does the analyst add 0.1N sodium arsenite solution OR ascorbic acid crystals until a drop of sample produces no color on indicator paper?	6.2				
<b>If Determining Cyanides Amenable to Chlorination:</b>					
5) Are two identical 500 mL aliquots prepared in order to determine amenable cyanide?	7.1.1, 7.1.2				
6) Is preparation performed under amber light, and are samples not exposed to U/V light, fluorescent light, or sunlight during processing?	7.1.1				
7) Is only <u>one</u> of the two sample aliquots carried through the following preparation to induce chlorination?	7.1.2				
8) Is calcium hypochlorite added to sample dropwise while agitating and maintaining the pH between 11 and 12 with 1.25N NaOH until an excess of chlorine is present as indicated by KI-starch paper turning blue? ( <i>Perform in a fume hood!</i> )	7.1.2				

**Notes/Comments:**

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9) Is the excess chlorine maintained using continuous agitation for one hour while rechecking sample with KI-starch paper?	7.1.3				
10) After one hour, is 0.1N sodium arsenite added in 1 mL portions until KI-starch paper shows no residual chlorine?	7.1.4				
11) Is an excess 5 mL portion of sodium arsenite then added and samples analyzed by EPA 9014 or 9213?	7.1.4, 7.1.5				
12) Is amenable cyanide determined by analyzing both sample aliquots for total cyanide and calculating the difference in the chlorinated and unchlorinated sample?	7.1.5				
<b>If Determining Total Cyanide:</b>					
13) Is a 500 mL sample aliquot placed into a boiling flask (or sample diluted to 500 mL)?	7.2.1				
14) Is a 50 mL portion of 1.25N NaOH added to the gas scrubber?	7.2.1				
15) Is a slow stream of air introduced into the boiling flask and the vacuum adjusted so that about two bubbles of air per second enter the flask through the air inlet?	7.2.2				
16) If samples were known or suspected to contain nitrate or nitrite, or if bismuth nitrate was added to samples, was 50 mL of 0.4 N sulfamic acid solution added through the air inlet tube followed by mixing for three minutes?	7.2.4				
17) Is a 50 mL portion of 18N H <sub>2</sub> SO <sub>4</sub> added through the air inlet, the inlet tube rinsed with water, and airflow allowed to mix the flask contents for three minutes?	7.2.5				
18) Is a 20 mL portion of 2.5M magnesium chloride added through the air inlet and the inlet tube washed with water?	7.2.6				
19) Is the solution heated to boiling and then refluxed for one hour?	7.2.6				
20) Is heat turned off and airflow continued for at least 15 minutes?	7.2.6				
21) Is solution transferred to a 250 mL volumetric flask and diluted to volume with water?	7.2.7				
22) Is total cyanide determined by EPA 9014 or 9213? <i>(Distillates may be stored at 4°C if not analyzed immediately.)</i>	7.2.8				
Notes/Comments:					

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<b>Quality Control:</b>					
23) Is at least one reagent blank carried through preparation with one per analytical batch or one in every 20 samples?	8.2				
24) Is at least one check standard carried through preparation with each batch of samples, with the result within 15% of the expected value?	8.3				
25) Are a duplicate and a spike analyzed with every 20 samples?	8.4, 8.5				
Notes/Comments:					